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ABLATIVE PERFORMANCE OF UNCOATED SILICONE-MODIFIED AND SHUTTLE BASELINE REINFORCED CARBON COMPOSITES

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16. Abstract

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SUMMARY

An investigation has been made of the relative ablative performance of uncoated silicone-modified reinforced carbon composite (RCC) and uncoated shuttle baseline RCC substrates. The test specimens were 13 plies (5.3 to 5.8 millimeters) thick and had a 25-millimeter-diameter test face. Prior to arc-tunnel testing, all specimens were subjected to a heat treatment simulating the RCC coating process. During arc-tunnel testing, the specimens were exposed to cold-wall heating rates of 178 to 529 kilowatts/meter^2 and stagnation pressures ranging from 0.015 to 0.046 atmosphere at Mach 4.6 in air, with and without preheating in nitrogen. The results show that the ablative performance of uncoated silicone-modified RCC substrates is significantly superior to that of uncoated shuttle baseline RCC substrates over the range of heating conditions used. These results indicate that the silicone-modified RCC substrate would yield a substantially greater safety margin in the event of complete coating loss on the shuttle orbiter.

INTRODUCTION

A silicon carbide-coated carbon-carbon composite, generally known as reinforced carbon composite (RCC), will be employed as the heat shield for the wing leading edge of the space shuttle orbiter. (See ref. 1.) These composites are manufactured by multiple impregnation of a carbon cloth with a carbon bearing resin. The silicon carbide coating is produced by thermally diffusing silicon into the outer layers and converting it to silicon carbide by reaction with the carbon in the host matrix. (See refs. 1 and 2.)

The current shuttle baseline RCC employs a polyfurfuryl alcohol (PFA) as the densifying impregnant. In the uncoated condition, this material has been shown to oxidize readily. (See refs. 3 and 4.) Also, arc-tunnel tests of coated shuttle baseline RCC have shown that substantial subsurface oxidation occurs because of cracking or crazing of the siliconized outer layer. (See refs. 5 to 8.) Thus, if the siliconized layer were damaged or lost in an actual flight, the substrate could ablate rapidly, perhaps causing a heat-shield failure.

Under NASA contract, Los Alamos Scientific Laboratory (LASL) undertook a preliminary investigation of methods to improve the oxidation resistance of the uncoated RCC substrate. In that study, thermogravimetric analysis up to 1020 K showed that the oxidation resistance of uncoated RCC substrates could be increased by as much as 60 percent by substituting a phenolic resin containing a silicone for the normally used PFA (ref. 9). The present study was undertaken to compare the ablative performance of uncoated silicone-modified RCC to that of uncoated shuttle baseline (PFA impregnated) RCC at surface temperatures and flow conditions representative of shuttle-orbiter reentry.

SYMBOLS

D	diameter, m
H_o	stagnation enthalpy, J/kg
$\dot{m}_s$	mass-loss rate per unit surface area, g/m^2-s
p_o	stagnation pressure, atm (1 atm = 101.3 kPa)
$\dot{q}_{cw}$	convective cold-wall stagnation heat-transfer rate, W/m^2
T	temperature, K
ϵ	emittance
τ	time, s

MATERIALS AND SPECIMENS

The manufacture of RCC begins with a prepregged graphite cloth which is laid up, molded, cured, and pyrolyzed. At this stage, the material is designated RCC-0. After three subsequent impregnation and pyrolysis cycles, the material is designated RCC-3. A detailed description of the fabrication process may be found in reference 1.

Samples of RCC-0 in the form of 13-ply (approximately 5.3- to 5.8-mm) thick bars about 150 mm long and 25 mm wide were supplied by Vought Corporation to LASL. These samples were processed through three impregnation-pyrolysis cycles as follows: The bars were impregnated with a silicone-phenolic resin at 343 K in nitrogen at 1.4-MPa pressure, cured in air up to 473 K over 71 hours, and pyrolyzed for 136.5 hours up

to 1175 K in 1-atm argon. The silicone-phenolic resin had been stripped of its solvent, isopropyl alcohol, prior to impregnation of the samples.

Arc-tunnel test specimens were machined from these bars to the configuration shown in figure 1(a). Arc-tunnel specimens of shuttle baseline material (RCC-3) in the configuration shown in figure 1(b) were supplied by Vought Corporation. The difference in specimen configurations was due to the inadequate width of the RCC-0 samples supplied to LASL. However, all specimens had a nominal 25-mm-diameter test face and had the same nominal 13-ply thickness.

After machining, all specimens were subjected to a final heat treatment (pyrolysis) in helium for 12 hours including approximately 3 hours at the maximum temperature, 2000 K. The purpose of this heat treatment was to simulate the thermal effect of the silicon carbide coating process used on RCC. (See ref. 1.)

For arc-tunnel exposures, the specimens were contained in a silicon carbide-coated bulk graphite holder shown in figure 2. Previous studies had shown that a very nearly uniform heating rate could be maintained across the specimen area by using a holder of this configuration.

APPARATUS AND TEST ENVIRONMENTS

The tests were conducted in a supersonic arc-heated tunnel, which is described in reference 10. For the present study, the apparatus was equipped with a conical nozzle having a 7-cm-diameter throat and a 22.9-cm-diameter exit. A radiometer operating in the 2.0- to 2.6-μm-wavelength region was used to monitor test-specimen surface temperature. The radiometer had been calibrated using the same geometry, window, and mirrors employed in the actual tests.

Two types of tests were conducted in this study. One type employed air as the test medium throughout the entire test. A second type employed nitrogen as the test medium to preheat the specimens without oxidation before converting the test medium to air for the duration of the test. The purpose of eliminating oxidation during the initial transient-heating portion of the tests was to provide a better correlation between mass-loss rate and specimen surface temperature.

During arc-tunnel testing, the specimens were exposed to cold-wall heating rates of 178 to 529 kW/m^2 and stagnation pressures ranging from 0.015 to 0.046 atm at Mach 4.6 in air. Specimen test conditions are given in detail in table I.

PROCEDURES

Prior to the tests, the desired flow conditions were determined and calibrated with respect to stagnation-point heat-transfer rate, stagnation pressure, power input to the arc heater, and tunnel mass-flow rate. The enthalpy values shown in table I were computed using the heat transfer and pressure measurements obtained in air after each test.

Tests in Air

After the arc tunnel was started, time was allowed for the flow conditions to stabilize. Heating rate and stagnation pressure were measured with a hemispherical calorimeter and a pitot probe, respectively. The specimen was then inserted into the test stream with the test face normal to the flow. Upon removal of the specimen from the stream, heating rate and stagnation pressure were measured again. The posttest measurements were used as the actual test conditions.

Tests in Air With Nitrogen Preheating

The tunnel was started, the flow was stabilized, and the flow conditions were measured in a nitrogen test stream. The arc heater was operated at the previously calibrated arc current, and the nitrogen flow rate was adjusted to give the desired arc chamber pressure. The preheat times were selected to allow the model surface temperature to approach that expected for the air portion of the test. The test medium was changed from 100-percent N_2 to 100-percent air with the transient effects lasting approximately 4 seconds. Flow conditions were again measured after the specimen was removed from the test stream, and these measurements were used as the actual test conditions.

Mass-Loss Determination

Mass loss was determined by weighing the specimens before and after arc-tunnel testing. Prior to initial weighings, the specimens were kept at room temperature in a dry storage cabinet containing a desiccant. After arc-tunnel exposure, specimens were allowed to cool to room temperature and were then weighed.

RESULTS AND DISCUSSION

Parallel arc-tunnel exposures were performed on uncoated specimens of silicone-modified and shuttle baseline RCC at a wide range of heating conditions representative of shuttle-orbiter reentry. Half of these tests were conducted completely in air, and half were conducted in air following a preheat period in nitrogen.

The relative ablative performance of the two materials was evaluated on mass-loss rate per unit surface area determined from initial front surface area and mass measurements before and after arc-tunnel exposure. The mass-loss rate per unit area, exposure time, and surface temperature experienced by each specimen are shown in table II.

The emittance of all specimens was assumed to be 0.8 in the 2.0- to 2.6-μm-wavelength band and to be independent of temperature. Preexposure room-temperature, spectral reflectance measurements made on several specimens of each material showed their emittance in this wavelength band to lie within the 0.7 to 0.9 range, which is generally accepted for this class of materials.

Representative surface-temperature histories for specimens tested with and without nitrogen preheating are shown in figures 3 and 4, respectively. The reason for the pronounced difference in the surface-temperature-rise rates of the two materials in the transient portion of the tests is not known. In figure 3, the rapid increase in temperature when the stream was converted from nitrogen to air is apparent. As can be seen in figures 3 and 4, the specimens tended to reach a quasi-steady surface-temperature condition before the completion of the test. The temperature of the silicone-modified specimens tended to oscillate throughout the later portion of the higher heating-rate tests. The surface temperatures reported in table II and used in following figures are representative of the quasi-steady portion of the tests.

Figure 5 shows mass-loss rate as a function of temperature for both materials tested. The significantly lower mass loss experienced by the silicone-modified RCC, ranging from 20 percent at 1300 K to 70 percent at 1700 K of the mass loss experienced by the shuttle baseline RCC, is evident. Nitrogen preheating did not have any apparent effect on the mass-loss rates of either material.

The mass-loss data have been normalized with respect to $p_o^{1/2}$, where p_o is the stagnation pressure, and plotted as a function of temperature in figure 6. Although there is some uncertainty about the precise value of the exponent of pressure, a wide range of values between zero and one having been reported in the literature, a value of 1/2 has wide acceptance. (See ref. 11.) Including the effect of pressure, the superiority of the silicone-modified RCC is still evident, with its mass-loss rate ranging from one-quarter to three-quarters that of the shuttle baseline material at temperatures from 1300 to 1700 K. However, figure 6 clearly shows that, at the higher temperatures, the performance of the two materials is approaching parity. Extrapolation indicates that parity should occur near 1800 K, the lower boundary of the diffusion-controlled oxidation regime for carbon. (See refs. 11 and 12.)

A comparison of the mass-loss rates of the two materials at each test condition is given in figure 7. The performance of the silicone-modified RCC is clearly superior to

that of the shuttle baseline RCC at every combination of heating rate and stagnation pressure. These results indicate that the silicone-modified RCC substrate would yield a substantially greater safety margin than the shuttle baseline RCC substrate in the event of complete coating loss on the shuttle orbiter. The results also indicate that, coupled with an equivalent coating, the subsurface oxidation of coated RCC could be significantly reduced by the substitution of a silicone-phenolic resin as the densifying impregnant in the manufacture of RCC substrates for the shuttle orbiter.

Photographs of typical specimens of shuttle baseline and silicone-modified RCC after testing are shown in figures 8 and 9. No unusual features are apparent in the posttest photograph of the shuttle baseline RCC (fig. 8(b)). However, the posttest photograph of the silicone-modified RCC shows a new, widely dispersed, white phase (fig. 9(b)). Figure 9(c) is a larger magnification scanning electron microscope (SEM) photograph of the specimen surface. This new phase is apparently a reaction product formed by the silicon from the impregnating resin. Energy dispersive X-ray analysis shows these white areas to be very rich in silicon; but because distinct X-ray diffraction patterns were not found, specific identification of the product as either SiC, SiO_2, or some mixture of these could not be obtained.

Figure 10 shows posttest photographs of several other specimens of silicone-modified RCC. The specimens exposed to relatively low heating rates show a light, widely distributed, powdery residue of reaction product on the surface. The specimens exposed to much higher heating rates, however, show a very heavy residue concentrated at isolated locations on the specimen surface. The heavy residue buildup around the periphery is attributed to air leakage between the specimen and the holder.

The reaction product is very friable, and only slight agitation was necessary to separate it from the specimen surface. Owing to the appearance of isolated islands of residue on the surface of the specimens exposed to the higher heating rates and the oscillating surface temperature in the later part of the higher heating-rate arc-tunnel runs (see figs. 3 and 4), an alternating growth and spallation of the reaction product on the specimen surface are postulated.

Such a mechanism for removal of the reaction product from the specimen surface, whether due to thermal expansion mismatch, aerodynamic shear, or otherwise, is also compatible with the trend for the mass-loss rates of the two materials to approach parity. As the heating conditions become more severe, the rapid, alternating removal of the protective reaction-product sheath would result in fresh unreacted surface being exposed. Because the amount of silicon present in the carbon matrix is quite small, the reduction in mass-loss rate afforded by it should be almost insignificant in the diffusion-controlled regime.

CONCLUDING REMARKS

An investigation has been made of the relative ablative performance of uncoated silicone-modified reinforced carbon composite (RCC) and uncoated shuttle baseline RCC substrates. Specimens of each material were tested in an arc tunnel at cold-wall heating rates of 178 to 529 kilowatts/meter^2 and stagnation pressures of 0.015 to 0.046 atmosphere. The performance of the two materials was compared on the basis of mass-loss rate per unit surface area. Prior to testing, all specimens had been subjected to a heat treatment simulating the silicon carbide coating process used on RCC for space-shuttle application.

The performance of the silicone-modified RCC is clearly superior to that of the shuttle baseline RCC at every combination of heating rate and stagnation pressure. Normalized with respect to stagnation pressure, the mass-loss rate of the silicone-modified RCC is only one-quarter that of the shuttle baseline RCC at 1300 K to three-quarters at 1700 K. The results also indicate that the ablative performance of the two materials should approach parity near 1800 K.

The lower mass-loss rate of the silicone-modified RCC is apparently due to the formation of a silicon reaction product during heating in air. Indications are that at higher heating rates, the reaction product intermittently spalls, exposing fresh unreacted surface. Such a mechanism supports the conclusion that the mass-loss rates of the two materials would approach parity in the diffusion-controlled oxidation regime.

On the basis of these results, the silicone-modified material would appear to offer a substantially greater safety margin in the event of complete coating loss on the shuttle orbiter. The results also strongly suggest that coupled with an equivalent coating, the subsurface oxidation of coated RCC could be significantly reduced by substitution of a silicone-phenolic resin as the densifying impregnant in the manufacture of RCC substrates for the shuttle orbiter.

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October 28, 1976

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TABLE I.- SPECIMEN TEST ENVIRONMENTS

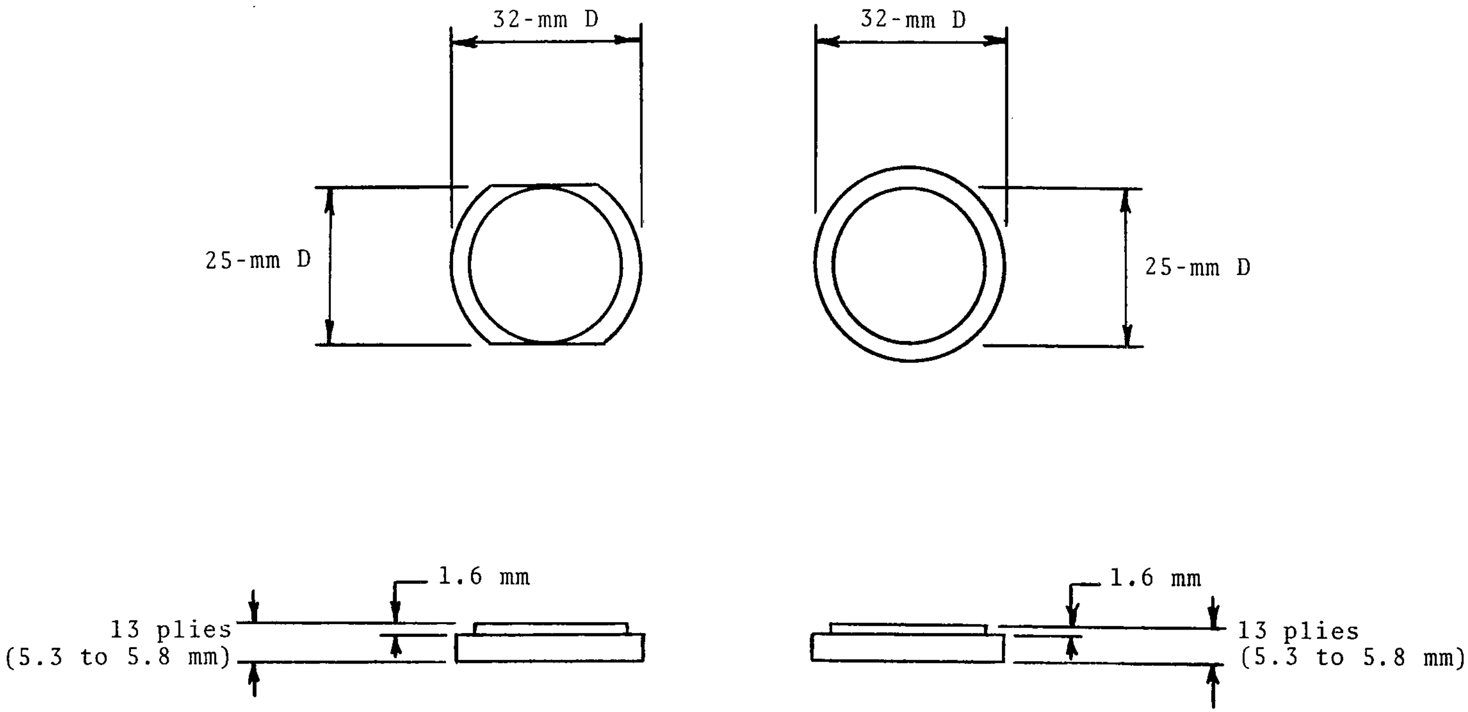
Specimen no.	Material	Nitrogen preheat	$\dot{q}_{cw}$, kW/m^2 (b)	p_o, atm	H_o, MJ/kg
A-100-3	STD[a]	No	178	0.020	3.44
A-100-1	↓	↓	236	.021	4.36
A-100-2	↓	↓	293	.042	3.88
A-100-4	↓	↓	506	.024	8.46
A-100-8	↓	Yes	197	.015	4.30
A-100-5	↓	↓	236	.022	4.25
A-100-7	↓	↓	511	.046	6.28
A-100-6	↓	↓	524	.024	8.79
LA-2-3	MOD[c]	No	192	.021	3.65
LA-2-1	↓	↓	246	.022	4.51
LA-2-2	↓	↓	293	.043	3.84
LA-2-4	↓	↓	506	.024	8.46
LA-7-4	↓	Yes	202	.015	4.39
LA-7-1	↓	↓	242	.022	4.39
LA-7-3	↓	↓	499	.043	6.32
LA-7-2	↓	↓	529	.024	8.88

[a]STD is shuttle baseline (polyfurfuryl alcohol impregnated) RCC substrate.
[b]Calculated from heat-transfer rate on hemispherical calorimeter.
[c]MOD is silicone-phenolic resin impregnated RCC substrate.

TABLE II.- TEST RESULTS

Specimen no.	Material	Nitrogen preheat	Air exposure time, s	Surface temperature, K	Mass-loss rate, g/m^2-s
A-100-3	STD[a]	No	300	1284	1.46
A-100-1			300	1412	2.89
A-100-2			240	1459	3.00
A-100-4			240	1727	7.22
A-100-8		Yes	300	1321	1.92
A-100-5			180	1469	2.88
A-100-7			180	1637	8.62
A-100-6			240	1741	7.97
LA-2-3	MOD[b]	No	300	1286	.44
LA-2-1			300	1444	.78
LA-2-2			240	1459	.99
LA-2-4			240	1686	3.95
LA-7-4		Yes	300	1326	.30
LA-7-1			180	1449	1.09
LA-7-3			180	1621	5.48
LA-7-2			240	1713	5.43

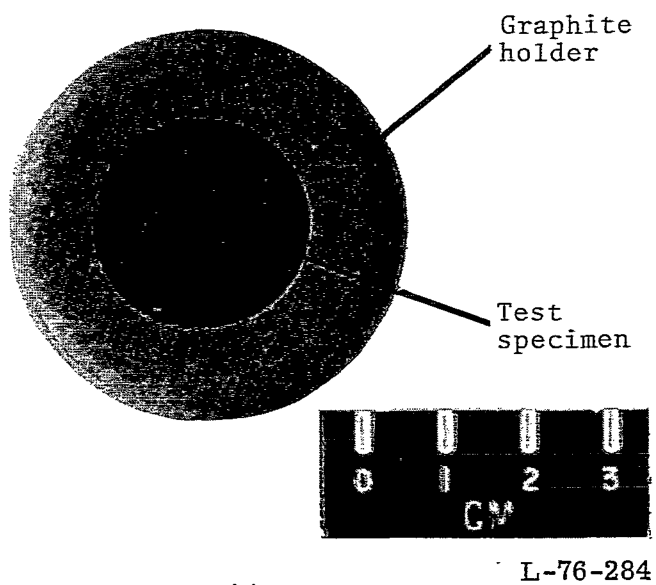
[a]STD is shuttle baseline (polyfurfuryl alcohol impregnated) RCC substrate.
[b]MOD is silicone-phenolic resin impregnated RCC substrate.



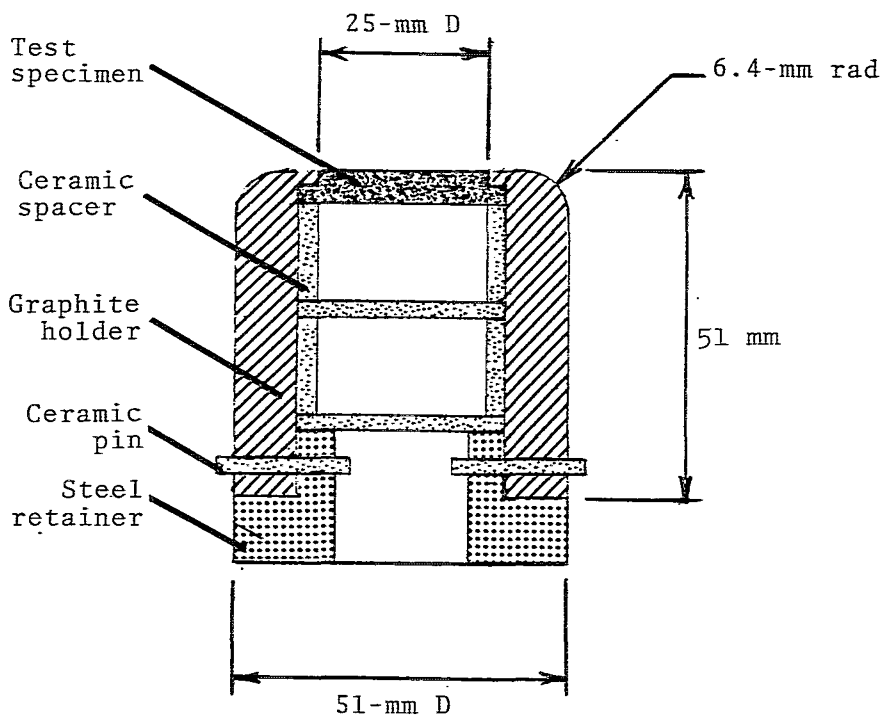
(a) Silicone-modified RCC specimen configuration.

(b) Shuttle baseline RCC specimen configuration.

Figure 1.- RCC specimen configurations for arc-tunnel tests.



(a) Top view.



(b) Cross-section side view.

Figure 2.- Specimen holder for arc-tunnel tests.

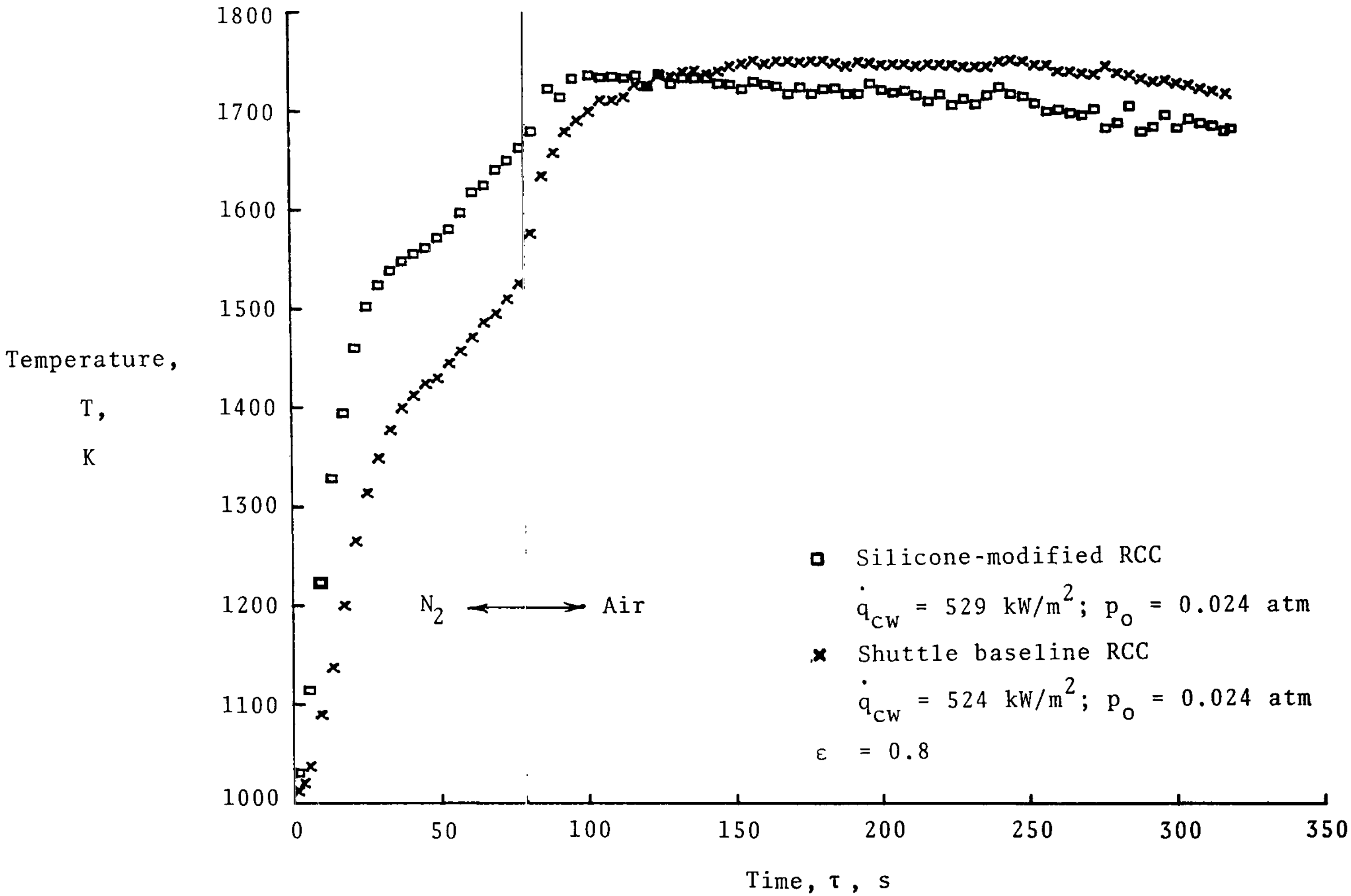


Figure 3.- Typical surface-temperature history for specimens tested with nitrogen preheat.

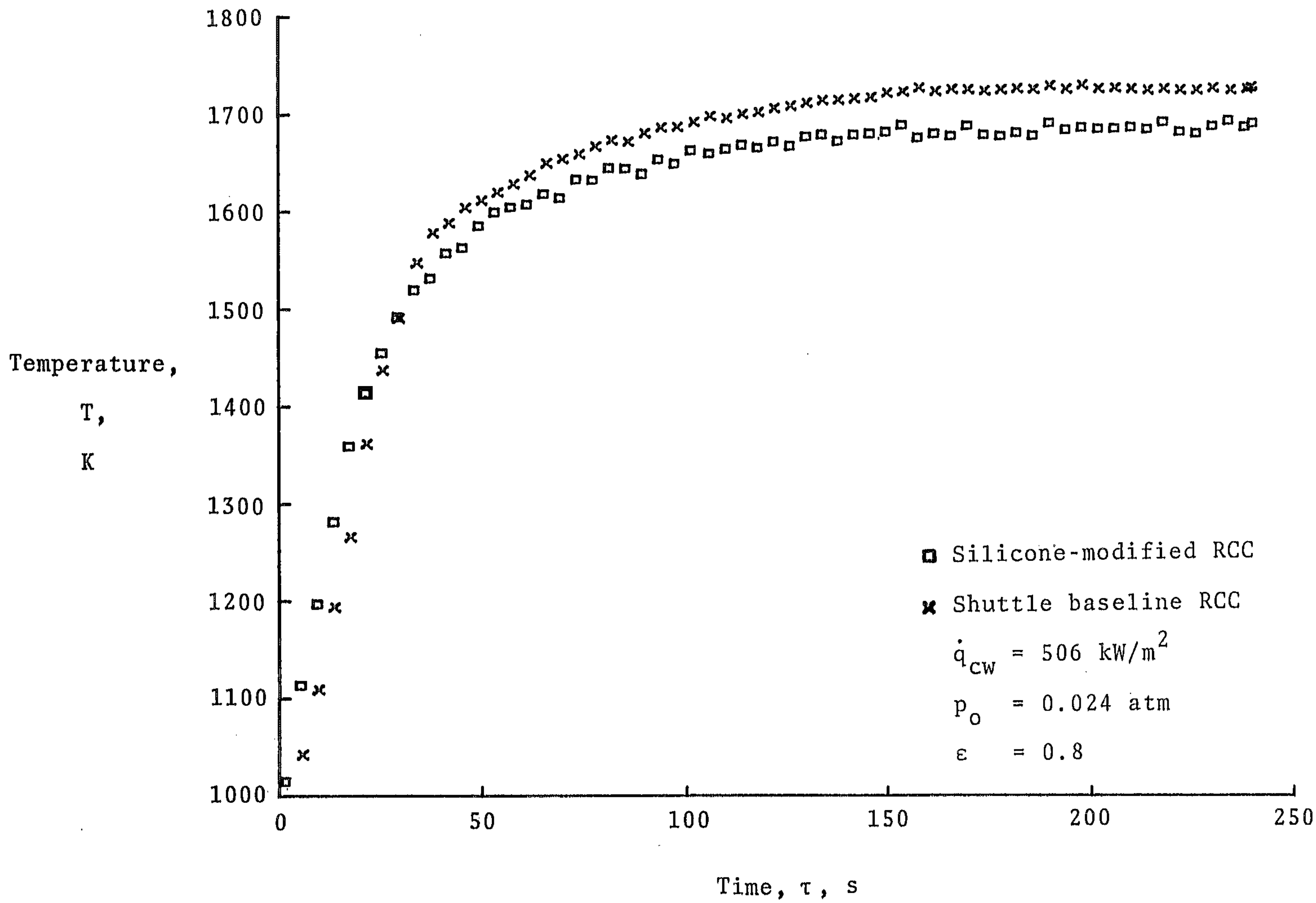


Figure 4.- Typical surface-temperature history for specimen tested in air only.

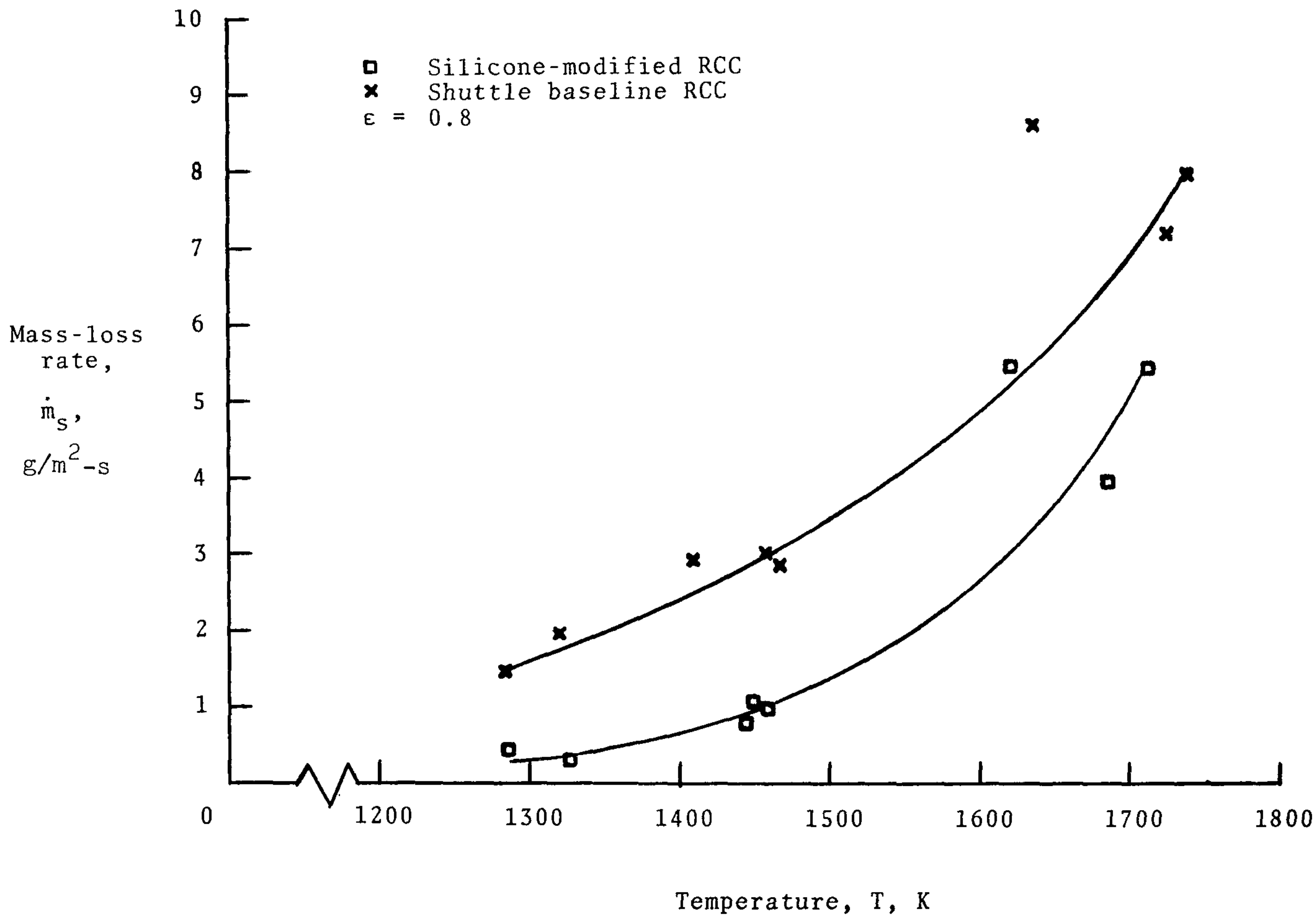


Figure 5.- Mass loss of RCC as function of surface temperature.

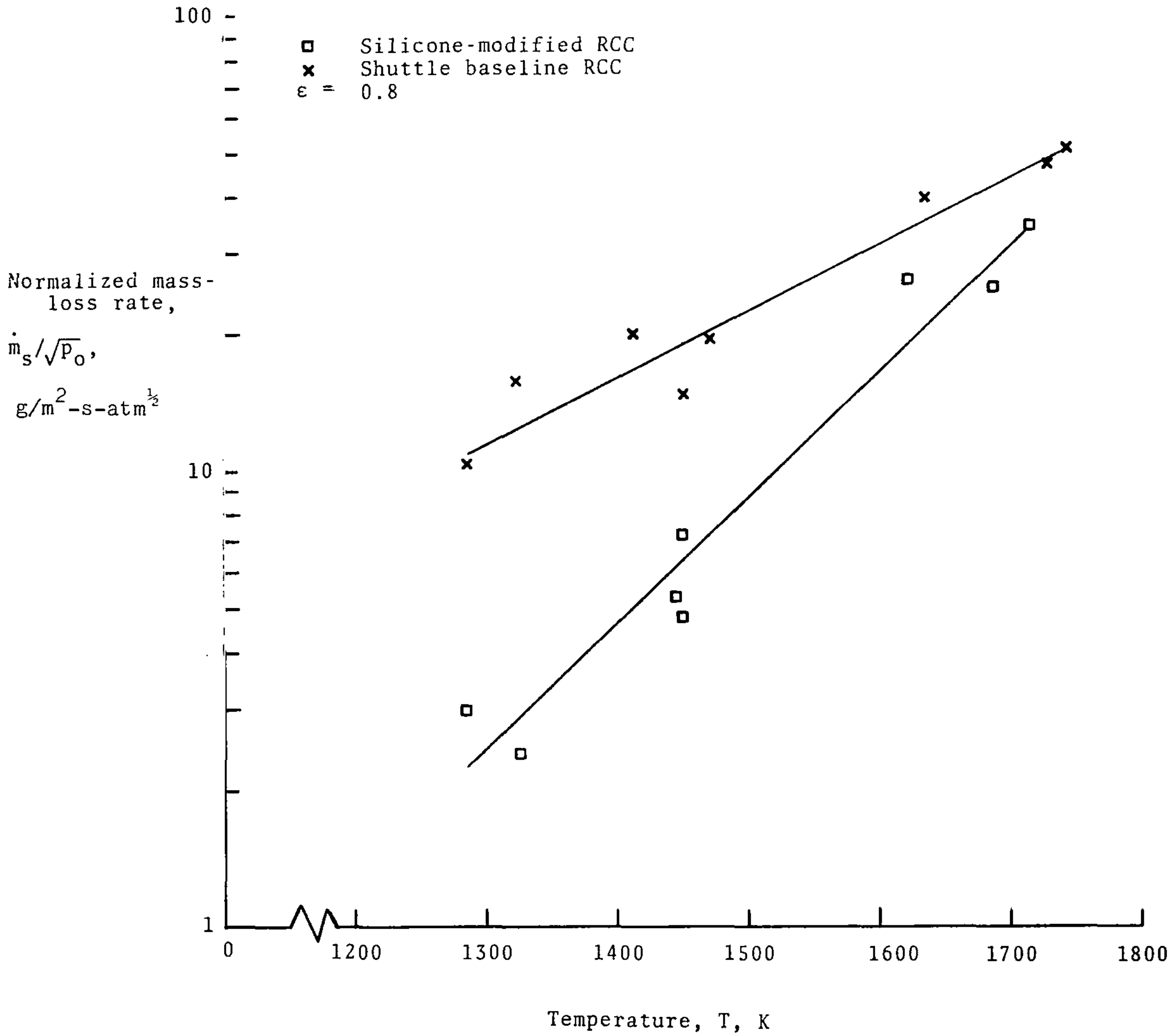


Figure 6.- Normalized mass loss of RCC as function of surface temperature.

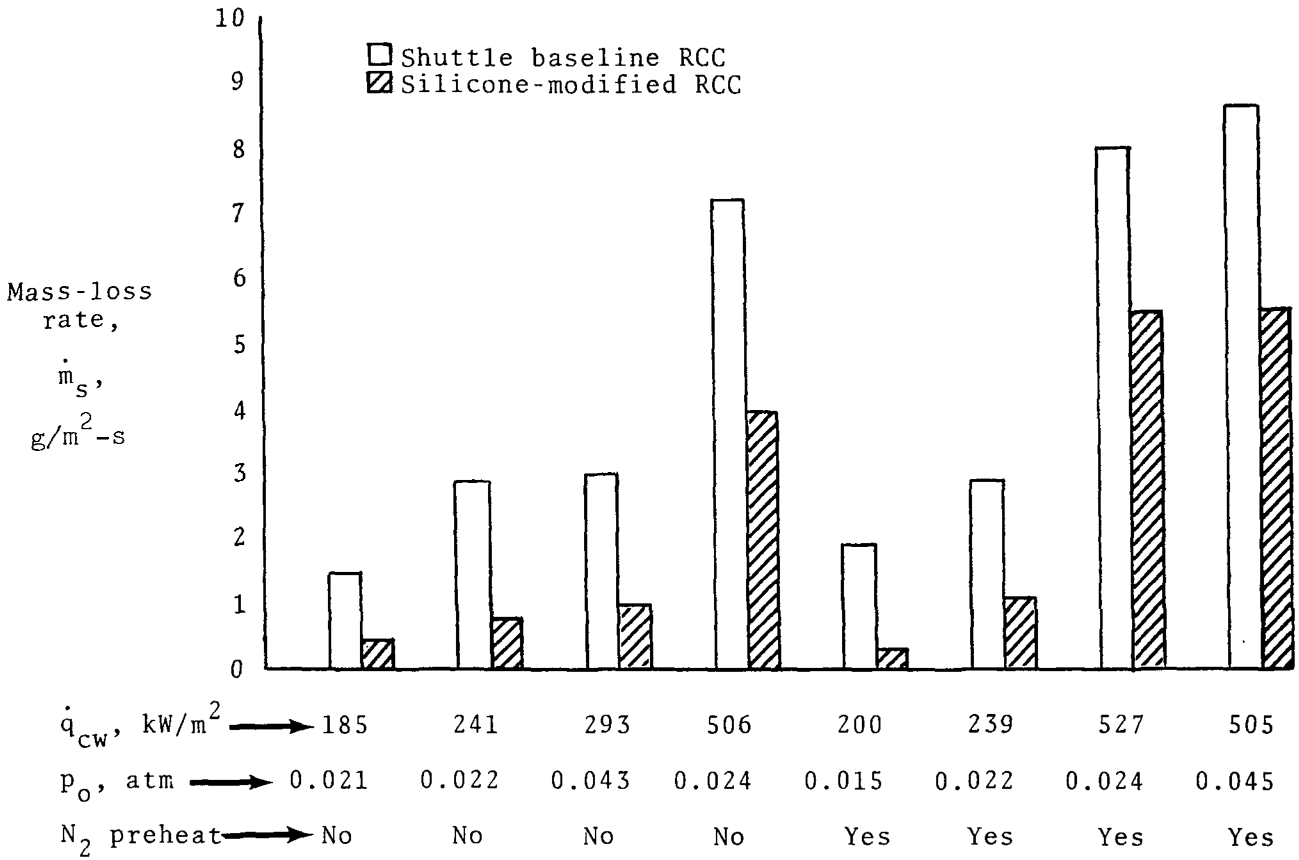
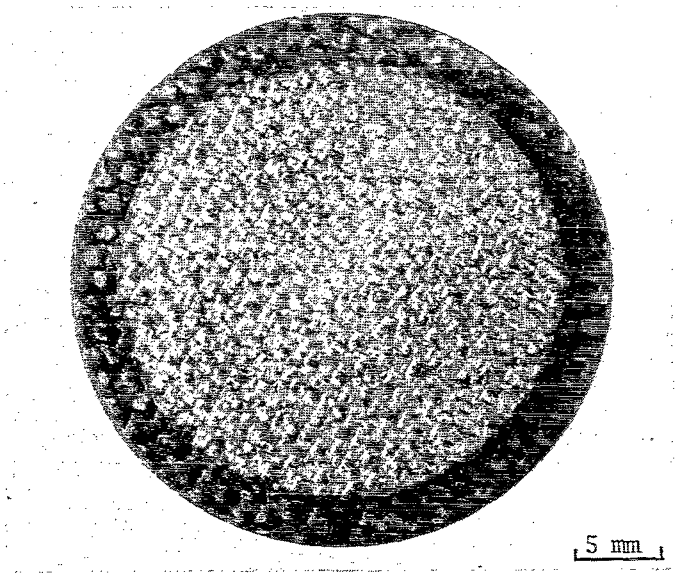
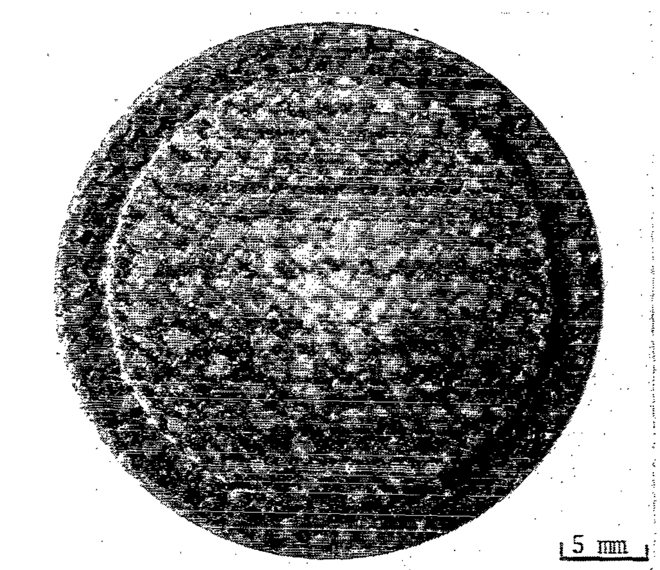


Figure 7.- RCC mass-loss comparison summary.



(a) Before testing.



(b) After testing.

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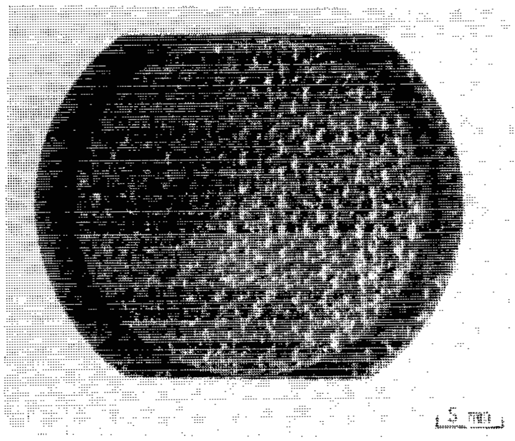
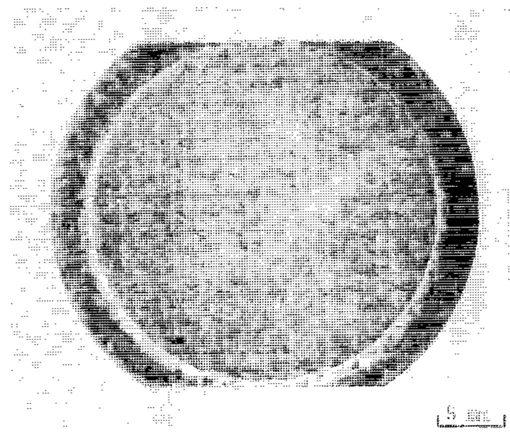
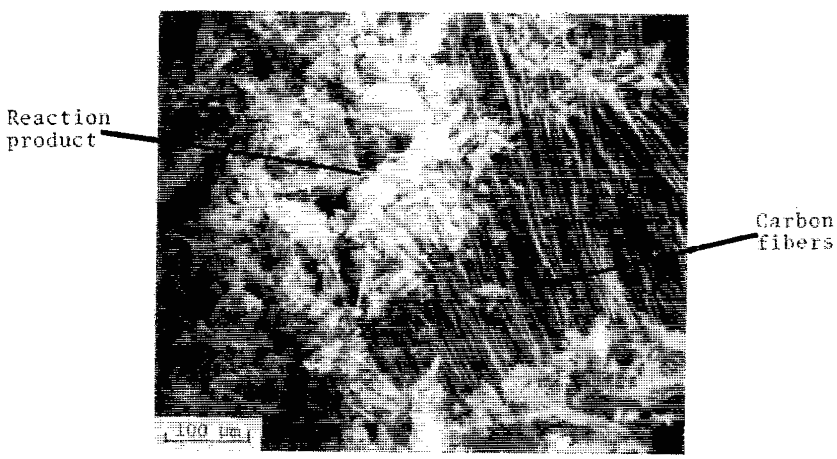
Figure 8.- Shuttle baseline RCC before and after testing.



(a) Before testing.



(b) After testing.



(c) SEM photograph of surface after testing.

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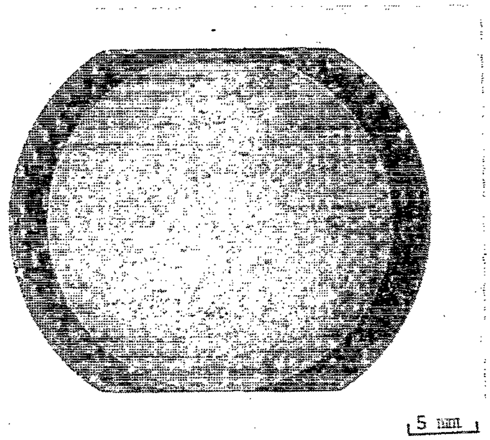
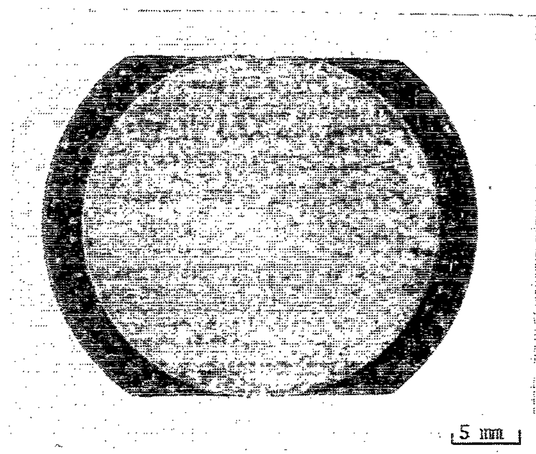
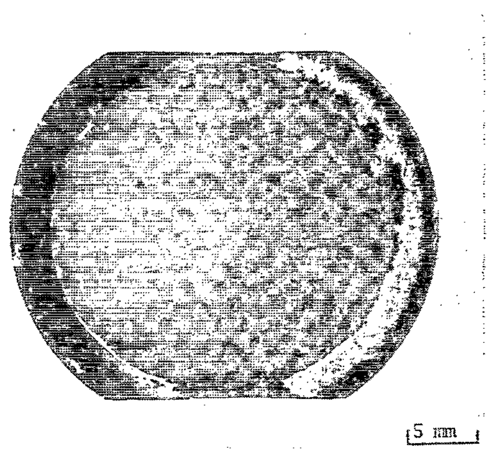
Figure 9.- Silicone-modified RCC before and after testing.



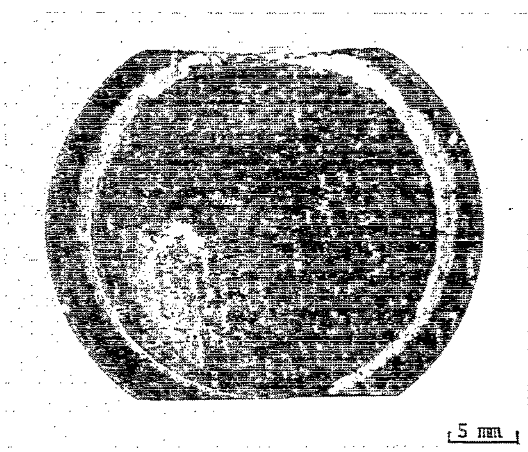
(a) $\dot{q}_{cw} = 192\ kW/m^2$; $p_o = 0.021$ atm.



(b) $\dot{q}_{cw} = 202\ kW/m^2$; $p_o = 0.015$ atm.



(c) $\dot{q}_{cw} = 506\ kW/m^2$; $p_o = 0.024$ atm.



(d) $\dot{q}_{cw} = 499\ kW/m^2$; $p_o = 0.043$ atm.

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Figure 10.- Silicone-modified RCC after testing.

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